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THE EFFECT OF TITRATABLE ACIDITY UPON HEAT COAGULATION IN EVAPORATED MILK

BY

REUBEN WALTER PETERSON
B. S. University of Illinois, 1917

THESIS

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
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I HEREBY RECOMMEND THAT THE THESIS PREPARED UNDER MY

SUPERVISION BY REUBEN WALTER PETERSON

ENTITLED THE EFFECT OF TITRATABLE ACIDITY UPON HEAT COAGULATION IN EVAPORATED MILK

BE ACCEPTED AS FULFILLING THIS PART OF THE REQUIREMENTS FOR

THE DEGREE OF MASTER OF SCIENCE IN DAIRY HUSBANDRY.

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*Required for doctor's degree but not for master's

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THE AUTHOR WISHES TO EXPRESS HIS SINCERE APPRECIATION TO PROFESSORS O. R. OVERMAN AND H. A. RUEHE FOR THEIR HELPFUL SUGGESTIONS AND KINDLY SUPERVISION IN THIS INVESTIGATION.

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THE EFFECT OF TITRATABLE ACIDITY UPON HEAT COAGULATION IN EVAPORATED MILK

I. INTRODUCTION

Milk has always, when fresh, an amphoteric reaction, turning blue litmus paper slightly red, and red litmus slightly blue. A similar reaction is possessed by certain phosphate solutions, and it is to the presence of such in milk that this reaction is due. This reaction has acquired a false importance, owing to the erroneous idea that neutrality as measured by the action of litmus is chemical neutrality; with the recognition of the fallacy of this idea the importance of the amphoteric reaction vanishes.

The acid reaction, however, has acquired some importance. It is well known that milk just drawn from the udder contains substances that cause it to appear to be slightly acid when examined by applying an acid test. This acidity of fresh milk is called the "apparent" acidity, to distinguish it from the "real" acidity due to lactic acid which is normally developed later by the action of bacteria on the milk sugar.

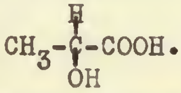
The apparent acidity is expressed in terms of lactic acid, because the latter is the acid common to milk. When it is stated that the apparent acidity averages 0.12 per cent, it means that by the method applied in measuring it, the result equals that which would be produced by the presence of 0.12 per cent of lactic acid; or, in other words, - the apparent acidity is expressed in terms of lactic acid.

The cause of the acid reaction of fresh milk to phenolphthalein has been the object of much investigation, which has resulted in narrowing down the probabilities to two or three sources, or a combination of them. Van Slyke and Bosworth (16) have presented good evidence to substantiate their theory that the apparent acidity is due to the acid phosphates present. Richmond (12)

attributes it partly to acid phosphates and partly to dissolved carbon dioxid. Bordas and Touplain (3) investigated the titratable acidity, with phenolphthalein, of milk serum, the coagulum containing the insoluble salts, the casein plus the insoluble salts, and the pure casein separated by the alcoholic method. They concluded that the original acidity is due to casein, and that no free acid or acid salts exist in the milk at the outset. Bordas (4) made further studies and reported that the increase in acidity is due to casein liberated from combination with calcium by the formation of calcium lactate.

This conflicting evidence as to the cause of titratable acidity in fresh milk is of less importance from the standpoint of factory practice than the question of the justice of an acid standard to the factory patrons.

The real or true lactic acid is formed by the action of certain lactic acid forming bacteria upon the milk sugar. By their action, the lactose is split up into lactic acid, almost quantitatively, a certain portion, however, being converted into other products, of which carbon dioxid is the most important. The micro-organisms which produce lactic acid are acted on inimically by acids, so that not much more than one per cent of lactic acid is usually formed.

According to Richmond (12), its formula is  $CH_3-\overset{H}{\underset{OH}{C}}-COOH$. It contains an asymmetric carbon atom; is not, however, optically active; but has a tendency to form compounds which contain less water. Lactic acid acts as a monobasic acid. It is not appreciably volatile in dilute solution, but passes over with water to a slight extent as the solution becomes concentrated. Lactic acid may be considered a normal constituent of milk, because almost immediately after milking milk contains organisms which produce lactic acid.

Total acidity is the apparent acidity supplemented by the development of real acidity, calculated as lactic acid. It is ordinarily estimated by titration with a standard alkali solution, usually sodium hydroxide, NaOH, using phenolphthalein as an indicator.

Van Slyke and Bosworth (16) point out that the acidity of milk should be estimated by titration after the addition of 2 c.c. of a saturated solution of neutral potassium oxalate to 100 c.c. of milk, to remove the calcium. The results are lower if this is done, and is due to the fact that while Na_2HPO_4 is neutral to phenolphthalein, $Ca_3(PO_4)_2$ is the insoluble neutral calcium salt. The difference between the acidity before and after the precipitation of the calcium is an index of the soluble phosphoric acid in the milk. This is not, however, taken into account in the factory method of determining acidity, so is not further considered here.

The titratable acidity, therefore, is the total acidity in the milk. If determined immediately after the milk is drawn, the total acidity is identical with the apparent acidity. After the lapse of a time sufficient to permit the bacteria to act upon the milk sugar, the total acidity includes both the apparent acidity and the real acidity. To find the percentage of real acidity the apparent acidity must be known, and subtracted from the total acidity.

The urgent necessity for some means of determining the quality of milk received at condenseries and other dairy manufacturing plants resulted in the use of the acid test as a measure of quality. This is a very rapid and simple procedure. An arbitrary standard of 0.18 per cent lactic acid was established, above which all milk was rejected. Objection has been raised to this standard because the percentage of apparent acidity may be higher than the standard used to reject the fresh milk which was normal in every other respect.

The percentage of apparent acidity that may be present is not constant for all milk. It varies similarly to the way the percentage of fat varies. McInerney (9) found the percentage of apparent acidity in fresh milk varied from 0.10 to 0.22 per cent.

If it is true that any considerable number of cows, or any one breed of cattle, consistently give milk which has a higher apparent acidity than the

standard maintained, or if the acid test fails to measure the quality of the milk at the receiving platform, the justice of such a standard necessitates further investigation.

The principal objection to milk high in acidity, in the case of the evaporated milk manufacturer, is that it cannot be "processed" without forming a curd that is difficult or impossible to break up by shaking. Whether apparent or real acidity produce the same effect has not been definitely proven.

The main object of this investigation is, therefore, to determine the effect of titratable acidity, - apparent or real - upon the heat coagulation in the process of sterilizing evaporated milk.

II. HISTORY: REVIEW OF PREVIOUS WORK

A. NOMENCLATURE

Considerable confusion exists in the nomenclature of concentrated milk products. Whereas most of such products are referred to by the public as condensed milk, technically, condensed milk is fresh whole milk from which a considerable portion of the water has been removed, to which cane sugar has been added, and, according to the United States Government standard, must contain not less than 8 per cent butterfat, and 28 per cent of total milk solids. Condensed milk keeps partly because of the effect of the boiling temperature used before condensing, and partly because of the inhibitory action of the cane sugar which is present in the concentrated milk to the extent of from 42 to 46 per cent, averaging about 44.5 per cent. It is usually guaranteed to remain in good condition for six months after leaving the factory.

Concentrated milk is milk from which a considerable portion of the water has been removed. It is not a sterile product, and keeps only a short time, even under refrigerated conditions. It is used mostly to reduce transportation charges in shipping, - as from a receiving station to a central factory. The percentage of total solids varies from 40 to 50 per cent.

Plain bulk condensed milk is whole or skimmilk which has been thickened by the application of live steam introduced directly into the concentrated milk. This is usually referred to as "superheating", and the product as "superheated" condensed. The condensation is not carried as far as in the case of concentrated milk. The finished product varies from 27 to 40 per cent total solids, and from 0.3 to 12 per cent butterfat. It is not sterile, and must be kept under refrigerated conditions. The ice cream trade furnishes the largest market for the plain bulk condensed milk.

Evaporated milk, with which we are herein primarily concerned, is whole milk from which a considerable portion of the water has been removed, and is preserved in hermetically sealed cans by the application of heat sterilization. According to the United States Government standard, it must contain not less than 7.80 per cent butterfat, and 25.50 per cent total solids. This product is also guaranteed to remain in perfect condition for six months after leaving the factory.

Condensed milk was first introduced about 1853 (8). A patent on this product was obtained by Gail Borden, in 1856, covering the concentration of milk under a vacuum, with or without sugar. Evaporated milk followed in 1884, Mr. John B. Meyenberg, a native of Switzerland, obtaining the patent in this country. Considerable difficulty was experienced at first in the manufacture of evaporated milk, due mostly to lack of knowledge of the physical and chemical properties of milk.

In order to appreciate the difficulties encountered in the manufacture of evaporated milk, it is necessary to understand the procedure used.

B. PROCESS OF MANUFACTURE OF EVAPORATED MILK.

In the preparation of evaporated milk great care is taken to use the best quality of milk. If the capacity of the factory is such that the milk cannot all be utilized at once, the milk is cooled in order to prevent further

development of acidity.

Preliminary standardization is usually applied to get the butterfat and solids-not-fat in the same proportion in the initial product as these are to occur in the finished evaporated milk.

The milk is heated in the forewarmer to a temperature varying from 190 to 212 degrees Fahrenheit, by means of steam introduced directly into the milk. The milk is often held at the higher temperatures from two to twenty minutes before condensing. The purpose of heating in the forewarmer is to destroy as many of the living organisms as possible, and to facilitate the condensing operation.

The concentration is usually carried on under a vacuum of about 24 inches, at a temperature of about 130° F. Steam coils inside the pan, and a steam jacket, furnish the heat medium. The vapors from the milk are condensed by a spray of water, and removed by the vacuum pump. A Beaumé hydrometer is used to indicate the proper concentration.

After being concentrated, the warm milk is homogenized to thoroly emulsify the butterfat. It is then cooled to below 45° F. The product is then standardized to the desired composition.

Before canning a trial sterilization is made, and the process modified from the standard procedure in order to meet the requirements of that particular batch of milk. The cans are usually automatically filled, sealed, and tested for leakers or lightweights.

Sterilizing, - often referred to as "processing" - is the most important part of the manufacture of evaporated milk. The purpose is to destroy all forms of micro-organisms, insuring the keeping of the milk indefinitely. It also serves to give the milk a heavier body, or viscosity, and a darker color, which may or may not be desirable. Exact control of time and temperature are imperative. Too long heat, or too high heat, produces a curdy or

"livery" body that cannot be removed by shaking.

The milk in the sealed cans is usually subjected to heat varying from 232 to 243° F., for from 13 to 35 minutes. A standard process is shown on page 21. Dry steam, under pressure, is ordinarily used. To insure uniform sterilization care is taken not to close the blow-off or vent valve completely until all the air pockets have been expelled from the sterilizer. The cans revolve end over end during the heating and cooling, but may be stationary during a portion of the time the milk is held at the highest temperature.

The cans are carefully cooled in the sterilizer. Mechanical shaking is applied if necessary to break up any slight curd that may have formed. After a period of incubation to detect swells, the cans are sorted, labelled, packed, and shipped.

From this statement of the general procedure used in the manufacture of evaporated milk it is evident that there are many factors that influence coagulation on heating in the sterilizer.

C. FACTORS INFLUENCING THE COAGULATION OF EVAPORATED MILK IN STERILIZING

All the factors influencing the coagulation of evaporated milk in sterilizing have not been determined. The possible influences may be classified as follows:

a. Factors under the control of the manufacturer -

1. Titratable acidity of the milk.
2. Temperature in the forewarmer.
3. Concentration of the milk after evaporation.
4. Pressure in the homogenizer.
5. Addition of sodium bicarbonate.
6. Temperature in sterilizing.
7. Time in sterilizing.
8. Shaking after sterilizing.

b. Factors not under the control of the manufacturer -

1. Composition and balance of the milk salts.

2. Hydrogen-ion concentration.

The above factors are not of the same relative importance, and in many cases they may be eliminated entirely.

a. Factors under the control of the Manufacturer

1. Titratable acidity of the milk - this factor is discussed under a separate heading, "D", following, - "Acidity of the milk as a factor in sterilizing".

2. Temperature in the forewarmer - it is generally understood by the trade that the higher the temperature in the forewarmer the greater the heat that may be applied in the sterilizer without coagulation. Milroy (10) has shown that heating milk to high temperatures precipitates part of the calcium; and Sommer and Hart (14) suggest "that this reaction probably explains the increased stability brought about by the high forewarming". Rogers, Deysher and Evans (13) found "that the effect of high forewarming in raising the coagulating temperature of evaporated milk is evidently due to precipitation of part of the calcium. Heating to 95° C. (203° F.) usually raises the coagulating temperature of the evaporated milk from 15 to 30° C. The effect of high forewarming may be increased by prolonging the heating".

Hunziker (8) states "in this heating the casein of the milk is somewhat changed. There occurs partial, though invisible, precipitation, and the higher the temperature to which the milk is heated, the more pronounced is this change. This change is desirable, because the casein thereby surrenders, to a limited extent, its power and tendency to form a firm curd in the sterilizer".

If the milk will not stand boiling, naturally it cannot be processed; but because it does not visibly coagulate on boiling, the evaporated

milk may not necessarily be successfully processed. However, the beneficial effect of high forewarming is often lost thru careless plant operation in not boiling the milk, or failure to appreciate its importance.

3. Concentration of the milk - the inability to secure uniform concentration, or exact standard composition, has been largely done away with by the use of rapid and accurate methods of controlling such composition. Sommer and Hart (14) state "differences in concentration account partly for the difference in coagulation of fresh milk samples". While this may be true of fresh milk, the concentration of the finished evaporated milk is no longer important because it is under the complete control of the manufacturer thru standardization.

4. Pressure in the homogenizer - Hunziker (8) states "when the homogenizing is done under relatively high pressure the resulting homogenized milk increases in thickness, and is more susceptible to the curdling action of the heat in the sterilizer". Since the pressure is usually uniform in the same plant, while the curdling tendencies are not uniform, this factor is not important, and may be eliminated.

5. Addition of sodium bicarbonate - the use of sodium bicarbonate, while not recommended, has been rather general. The so-called baking soda, $NaHCO_3$, acts somewhat as a neutralizer of the acid, forming sodium carbonate upon boiling with water. This factor was eliminated in this investigation by using no soda.

6. Temperature in sterilizing - a difference of only one degree in temperature in the sterilizer may cause the milk to coagulate. Therefore, it is most important that the exact temperature be maintained. It is also essential that as high a temperature as possible be used, without causing curd to appear, in order to render the milk completely sterile.

7. Time in sterilizing - an exact time is as essential as an exact

temperature. The time taken to reach that temperature must be as uniform as possible. One minute too long, at the high heat, may cause curd to appear. It is generally assumed that one degree is equivalent to about one minute in sterilizing effect. The trial run determines the temperature and time that may be successfully applied at first. Variation must be made from one sterilizer batch to another to allow for changes in the same milk, but usually this will not be necessary.

8. Shaking after sterilization - mechanical agitation may remove a very fine curd, in from five to sixty seconds. It is usually unnecessary, however, and adds to the cost of handling the product. Few successful processes require shaking after sterilizing.

b. Factors not under the control of the manufacturer

1. Composition and balance of the milk salts - Sommer and Hart (14) state "the main factor in the heat coagulation of fresh milk is the composition of the milk salts. Apparently the casein requires a definite optimum calcium content for its maximum stability. The calcium content of casein is largely controlled by the magnesium, citrates, and phosphates present".

Experiments with dairy products at Wisconsin (15) resulted in the conclusion that "coagulation (in condensed milk) is due primarily to an excess of calcium salts, and any tendency to coagulate can be readily corrected by the addition of suitable citrates or phosphates".

Rogers, Deysher and Evans (13) found "the composition of the salts of milk is without influence on the temperature at which the evaporated milk curdles. It is evident, however, that whatever influence the acid-base ratio may have, it is usually obscured by other factors".

These results tend to show that the composition and balance of the milk salts, while a factor in fresh milk coagulation, are without appreciable effect on the heat coagulation of the evaporated milk in the sterilizer.

In addition such analyses could not be completed before the milk had been thru the entire plant process, hence this factor is not under the control of the manufacturer.

2. Hydrogen-ion concentration - it is very difficult to separate the effect of the hydrogen-ion concentration from that of the composition of the salts of the milk. Any change in the latter changes the hydrogen-ion concentration. When the milk is concentrated there is a uniform decrease in the pH (according to the Sorenson scale); in other words, an increase in the acidity.

Sommer and Hart (14) conclude that "hydrogen-ion concentration is not the determining factor in fresh milk coagulation. It is nevertheless a factor in fresh milk, and in commercial milks it may become an important factor".

Rogers, Deysher, and Evans (13) obtained results showing "the hydrogen-ion concentration, determined electrometrically, did not indicate that the relation between the acidity of the fresh milk and the coagulating point after it is evaporated is sufficiently uniform to make it of much value in grading milk".

The hydrogen-ion concentration, while an indication of true acidity would therefore have relatively little weight in determining the heat coagulability of the milk, even if it were more under the control of the manufacturer than at the present time.

D. ACIDITY OF THE MILK AS A FACTOR IN STERILIZING

The problem of the acidity of the milk as a factor in sterilizing resolves itself naturally into two divisions, - first, what is the amount of milk received at a factory containing over 0.18 per cent acid due to initial apparent acidity or true acidity?; and second, what is the correlation between varying amounts of titratable acidity and the heat coagulation of evaporated milk in sterilizing? The use of the alcohol test, in conjunction with the acidity test, also requires some consideration.

a. Apparent acidity in fresh milk

Rice (11) states "that individual cows were found giving milk with apparent acidity as high as 0.22 per cent", but fails to give the proportion of such milk found.

Sommer and Hart (14) found "titratable acidity directly from the cow varied from 0.102 to 0.257 per cent. Out of 86 samples, 45 are above 0.18 per cent, or about 51.2 per cent of the total samples".

McInerney (9) studying apparent acidity, according to breeds, obtained the following results -

"1. The per cent of apparent acidity in fresh milk varies from 0.10 to 0.22 per cent:

BREED	ACIDITY		
	Average	Highest	Lowest
Ayrshire	0.123%	0.150%	0.105%
Holstein	.136	.180	.100
Guernsey	.139	.160	.120
Jersey	.162	.220	.130
Shorthorn	.180	.200	.165

2. There appears to be no relationship between the period of lactation and the per cent of apparent acidity.

3. There appears to be no relationship between the age of the cow and the apparent acidity.

4. The kinds of feed had no effect on the percentage of acidity found in the fresh milk.

5. Fresh milk which showed a high percentage of solids-not-fat, and a high percentage of ash, showed a high percentage of apparent acidity.

6. Fresh milk comparatively low in solids-not-fat, and in ash, showed a lower percentage of apparent acidity."

Brew (5) found Holsteins varied from 0.12 to 0.16 per cent; and Jerseys and Guernseys from 0.16 to 0.25 per cent acidity.

In contrast to these results is the work of Fleming and Nair (7). "The normal acidity of fresh milk of individual cows is from 0.14 to 0.145 per cent. No milk from 18 individual Jersey and Holstein cows recorded showed over 0.17 per cent acidity at milking. The cows farthest along in the period of lactation showed the lowest acidities. There is no consistent dependence of high acidity upon high solids-not-fat content. The normal acidity of composite herd milk as received at the factory is 0.14 per cent. Only 0.26 per cent of the 766 herds tested, representing 7700 cows, furnished milk in October which would have been rejected on the basis of an apparently high acid condition, provided the milk was properly and sanitarily handled".

This conflicting evidence on the extent of apparent acidity calls for further study in connection with its effect upon the heat coagulation of evaporated milk.

b. Effect of varying amounts of acidity upon heat coagulation

Brew (5) states "the principal objection to milk high in acidity is that it cannot be processed without forming a curd that is difficult or impossible to break up by shaking. This is true in the case of milk high in real or lactic acid, but not true of milk merely high in apparent acidity".

Sommer and Hart (14) show "in fresh milk there is no direct relation between titratable acidity and heat coagulation. Acid fermentation in milk lowers the coagulating point, by changing the reaction, and lowering the citric acid content. However, the titratable acidity of fresh milk samples varies so widely that it is impossible to determine the extent of acid fermentation by titration. Therefore, it is impossible to use the acidity of milk as a criterion of coagulability".

Rogers, Deysher, and Evans (13) carried the results thru to the finished evaporated milk, and state "the variation in the coagulation temperature of mixed herd milk is comparatively small, and has little relation to the coag-

ulating temperature of the same milk after evaporation. Consequently, the temperature at which evaporated milk made from the mixed milk of a number of cows will curdle cannot be predicted with any accuracy by determining the coagulating temperature of the raw milk". They further conclude " that a titratable acidity of 0.19 to 0.20 per cent in a large batch of milk would almost certainly indicate an increase in acidity from the normal due to fermentation; the same titration in the milk from a small herd would be less conclusive; the acidity of milk from individual animals is subject to considerable variation, and the value of the acidity determination becomes very questionable. In any case, it is evident that while the effect of increased acidity brought about by bacteria has a marked effect on the coagulating point, there are other factors of equal or of even greater importance which may obscure the effect of increased acidity".

Experiments at Wisconsin (15) state "it was found that the titratable acidity of milk is not an indication of its coagulability when sterilized, and that condenseries are not justified in refusing milk of high acidity".

Dahlberg and Garner (6) found "the acid test as ordinarily used will reject a portion of the unsatisfactory milks, but as a whole is unreliable and inadequate as a means of determining the quality of milk for condenseries where evaporated milk is manufactured". The procedure outlined, however, fails to show the very important factor of temperature used in forewarming. It is presumed that the temperature was sufficiently high, and uniform, in all cases.

An inspection of their data shows that at Factory B the following results were obtained:

Batch number	No curd	Curdy
1	0.14% acidity	
2		0.175% acidity
3	.15	
4		.175
5	.165	
6		.175
7	.145	
8		.16

This would tend to indicate that the higher acidity might be an index of the coagulability of the milk during sterilization, as the higher acidity samples formed curd.

The other important factors having been controlled or eliminated, an attempt is made in this investigation to determine the correlation between varying amounts of acidity in the fresh milk and the heat coagulation in the evaporated milk.

c. Relative value of the alcohol and acidity tests of milk for condensing.

The alcohol test has been used to a limited extent for determining the quality of milk with special reference to its sanitary condition. This test is usually made by mixing equal volumes of milk and 68 to 75 per cent alcohol, and observing whether a coagulation results.

Auzinger (1) found "great fluctuations in the alcohol test (70%) with milk from single cows. Occasionally milk from the same cow gave a positive test in the morning, and not in the evening, or vice versa. The test might be positive one day, and not the next; and sometimes the first and last milk drawn showed fluctuations in the alcohol test". He concludes "that the alcohol test in normal milk from a single cow is independent of the acidity, and when the test is positive it is caused by a change in the milk salts, especially the calcium, in their relation to the milk proteins".

Ayers and Johnson (2) found "that while the addition of acid phosphates to milk increased the tendency to coagulate with alcohol, the neutral or dibasic phosphate had the opposite effect. The alcohol test becomes positive when an appreciable amount of acid is produced in the milk, but there is no consistent relation between the alcohol test and the total number of bacteria present, and is not a reliable index of the sanitary quality of the milk".

Dahlberg and Garner (6) show "that there is no direct relation between the coagulation of the milk with 75 per cent alcohol and the acid content of

milk as measured by the titration method; but milks high in titratable acidity as a result of fermentation will in the large majority of cases show coagulation with alcohol. The alcohol test shows good possibilities as a practical and reliable test for determining the quality of milk for condenseries making evaporated milk". In their study, as stated before, they fail to show the temperature used in the forewarmer. Their results were not conclusive, in that many samples in Factory A which did not coagulate with alcohol did coagulate in the sterilizer.

Since the value of the alcohol test has not been thoroly demonstrated, this test was applied together with the titratable acidity test, to determine the suitability of milk for the manufacture of evaporated milk.

III. PLAN OF THE INVESTIGATION

A. APPARATUS

The milk samples were forewarmed in a can by introducing live steam directly into the milk, similar to factory practice.

A small vacuum condensing unit was assembled, as shown in the photographs on page 17. A round pyrex flask, with a capacity of ten liters, served as the milk retort. A thermometer, blow-down or air inlet tube, milk inlet tube, and distilling head were fitted to the neck of this flask. A large "Liebig" type external water condenser was used to condense the vapors from the milk. A two liter suction flask retained the water removed from the milk.

A vacuum of about 73 centimeters, as measured on a mercury manometer, was created and maintained by means of a fine spray type of water suction pump.

Heat was applied by means of a boiling water bath around the lower portion of the flask.

A Beaumé hydrometer was used to indicate the approximate concentration. All analyses were made on the Mojonnier Tester. After standardization, the evaporated milk was homogenized, cooled, sealed in 16 ounce or talls cans, and

sterilized.

The sterilizer, shown on page 18, is a standard Fort Wayne sterilizer, containing two trays. The cans revolved in the cage end over end, at a speed of about 14 revolutions per minute.

Figure 1.

VACUUM CONDENSING UNIT

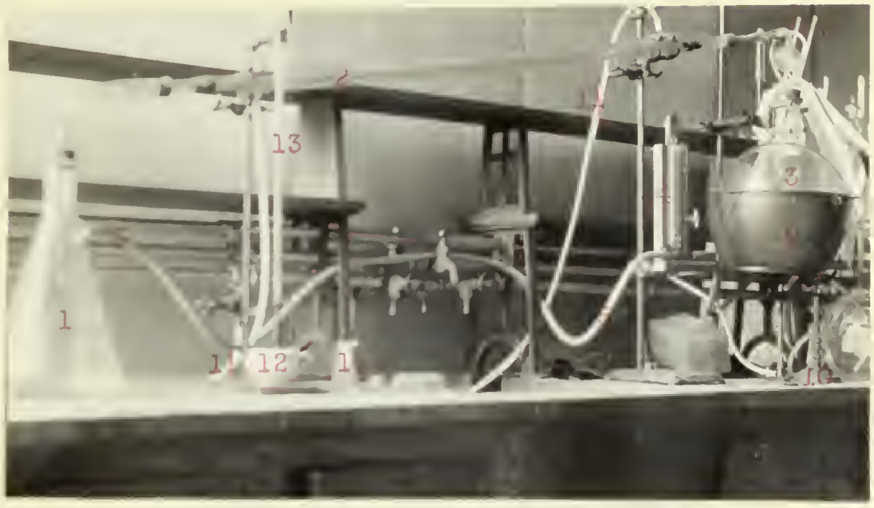
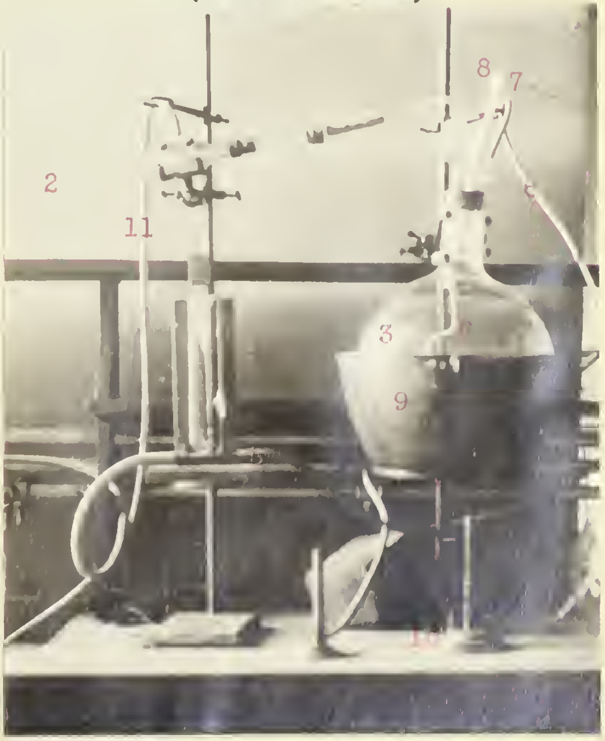


Figure 2

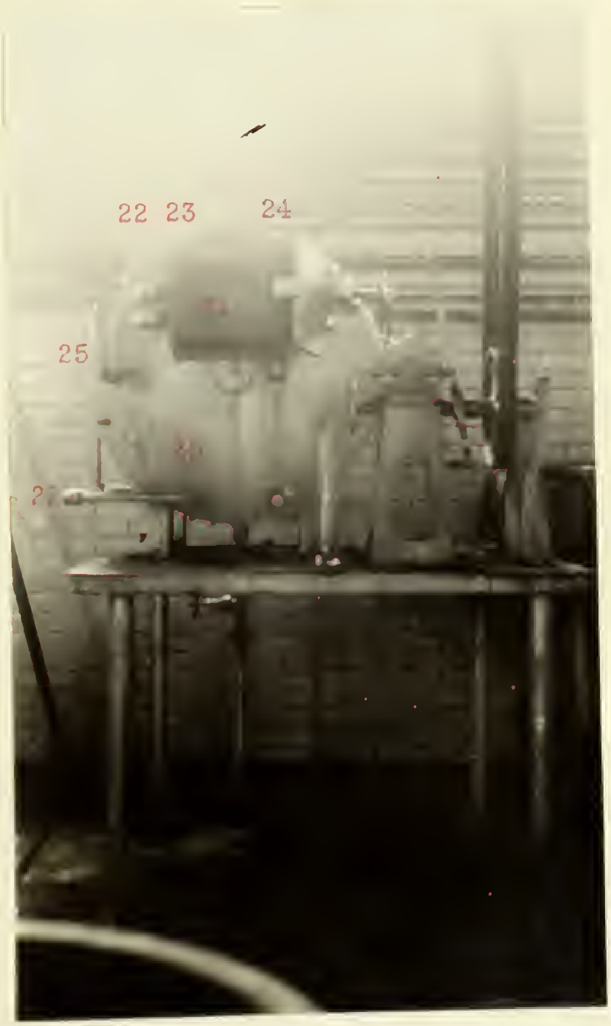
CONDENSING RETORT
(Detailed view)



1. Receiving flask, distillate.
2. External water condenser.
3. Glass retort, containing milk.
4. Manometer, measuring vacuum.
5. Vacuum produced thru water suction filter pump.
6. Milk intake from forewarmer.
7. Blow-down, or pop valve.
8. Thermometer.
9. Water bath.
10. Bunsen burner.
11. Water inlet and outlet, for Liebig condenser.
12. White cup for acid titration.
13. Burette for alkali.
14. Phenolphthalein, indicator.

Figure 3.

STERILIZER



20. Sterilizer.
21. Revolving cage.
22. Steam inlet, and valve.
23. Water valve, and inlet.
24. Pressure guage; safety
    valve located behind.
25. Thermometer.
26. Drain valve.

B. PROCEDURE

a. Test Methods Used.

1. Titration, - all milk samples were titrated according to the standard procedure. Decinormal alkali, N/10 NaOH, was used to neutralize the acid in the milk. 17.5 cubic centimeters (18 grams) of the milk sample were measured into a white cup. An equal volume of distilled water was added as an aid in distinguishing the end point. Three drops of Phenolphthalein were used as an indicator. The alkali was added until a faint pink color persisted for about thirty seconds.

The same procedure was used for the concentrated milk, except the pipette was marked to deliver exactly 18 grams of the standardized evaporated milk before sterilization.

2. Alcohol test, - 1 cubic centimeter of 75 per cent ethyl alcohol (Specific Gravity 0.8556) was added to 1 cubic centimeter of the milk. The extent of coagulation was noted after shaking.

3. Boiling test, - 3 cubic centimeters of milk were boiled in a test tube, and the extent of coagulation noted.

4. Butterfat and total solids, - all analyses were made by the Mojonnier method.

b. Preparation and care of samples.

Milk was secured directly from individual cows at the University Dairy Herd. This herd consists of pure bred Holsteins, Guernseys, Jerseys, Ayrshires, and some high grade cows. Samples of milk were also taken at the receiving platform from the patrons of the University Creamery, consisting of mixed herd milk from different farms.

When it was necessary to modify the milk in order to place the butterfat and solids-not-fat in the same proportion as these are to occur in the finished product, a portion of that sample was skimmed, and the standardization

accomplished with the skimmilk or cream from that individual sample. In this manner the influence of outside milk was eliminated.

The samples were titrated immediately after milking, in the case of individual cows. At the receiving platform samples were taken as soon as the milk was received in the morning. In all cases when it was necessary to hold samples for any length of time without change in acidity they were immediately cooled and stored at 32° to 35° Fahrenheit. Incubated samples were held at room temperature, 70° to 85° Fahrenheit, until the proper acidity developed.

c. Condensing.

The procedure outlined on page 6 for the manufacture of evaporated milk was followed as far as possible.

Preliminary standardization was practiced in order to get the butterfat and milk-solids-not-fat in the exact ratio they were to occur in the finished evaporated milk.

The milk was heated to a boiling temperature by introducing live steam directly into the milk, and not held, unless otherwise indicated.

The condensing was carried on under a vacuum of about 73 centimeters as measured on the mercury manometer, at a temperature of about 45° to 55° Centigrade (113° to 131° F.). Sufficient concentration was secured by a reading of 7.0° Beaumé, at 120° Fahrenheit.

The concentrated milk was standardized to the United States Government requirements of 7.80 per cent butterfat, 17.70 per cent solids-not-fat, and 25.50 per cent total solids; homogenized, and cooled.

The product was sealed in standard 16 ounce tall cans, and sterilized by the following process.

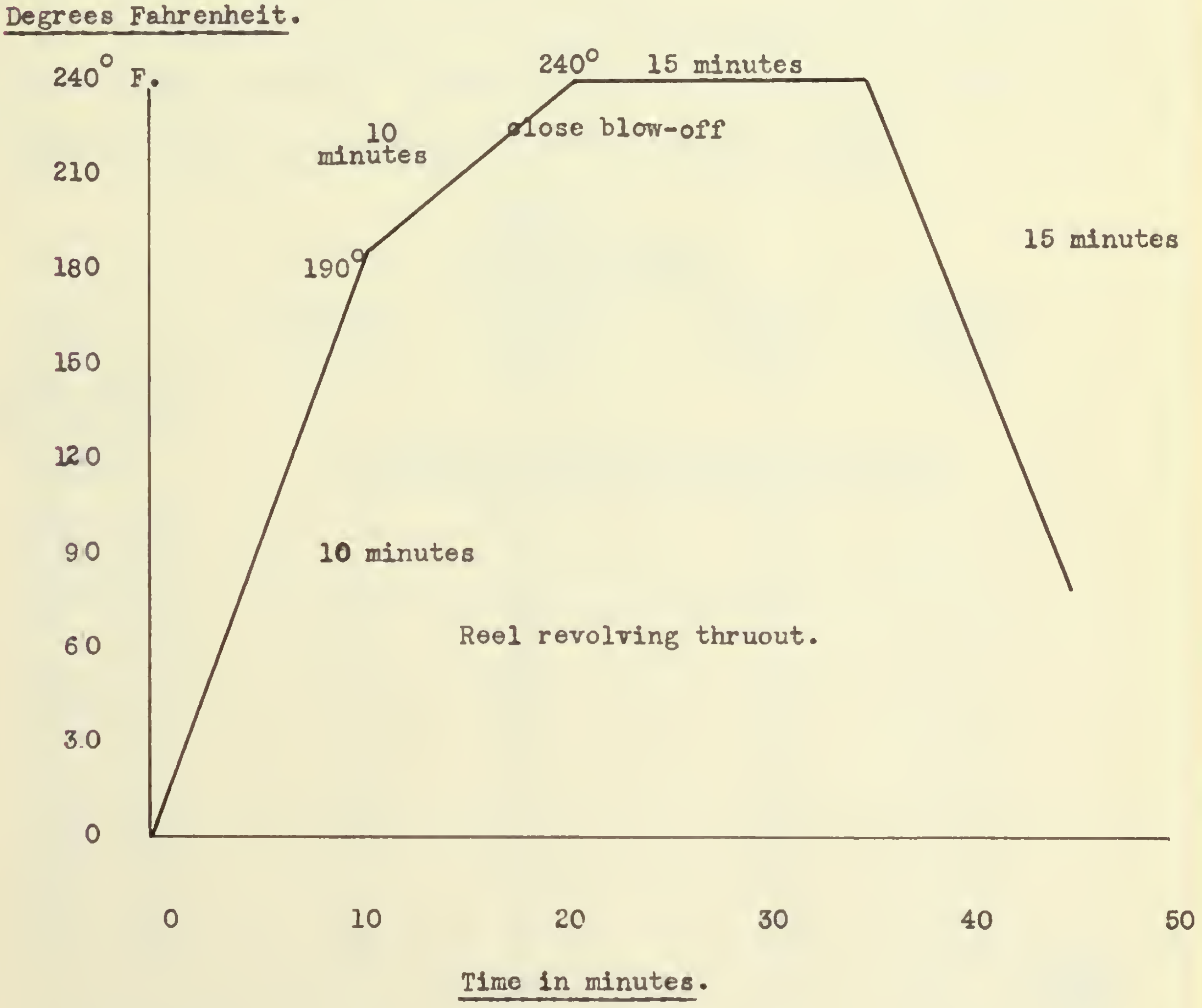
d. Method of processing or sterilizing.

Ten minutes was taken to reach or hold the heat at 190° F. From 190° to 240° F., the temperature was raised uniformly five degrees every minute, or

a total of 10 minutes more; making 20 minutes to reach 240° Fahrenheit. The blow-off or vent valve was closed at 220° Fahrenheit, 16 minutes after starting. The milk was held 15 minutes at 240° Fahrenheit, and then cooled to room temperature in about ten minutes. The reel was revolving thruout the entire process, at a speed of about 14 revolutions per minute. The milk was examined with and without shaking to note the formation of a curd.

Figure 4.

METHOD OF PROCESSING OR STERILIZING



IV. EXPERIMENTAL

Experiment 1.

APPARENT ACIDITY.

Milk samples were secured directly from the entire herd, consisting of representative cows of the four principal breeds. The series covered a period of about ten months.

Titratable apparent acidity was determined immediately, or the sample was cooled and held at 32° to 35° Fahrenheit. The average for each breed was obtained by using the weighted values.

A composite sample of all the milk from each breed, together with a butterfat, solids-not-fat, and total solids determination, is given at the end of Table 1. Table 1 is a compilation of the results obtained.

Table 1.

APPARENT ACIDITY

BREED:	AYRSHIRE	HOLSTEIN	JERSEY	GUERNSEY	
Number of cows represented	7	19	12	13	
ACIDITY	Number of samples of the given acidity				
.09 - .10%	2	0	0	1	
.10 - .11	5	4	1	0	
.11 - .12	3	7	0	1	
.12 - .13	1	8	3	0	
.13 - .14	6	12	4	2	
.14 - .15	3	15	5	1	
.15 - .16	3	12	12	4	
.16 - .17	3	8	4	10	
.17 - .18	4	2	5	11	
.18 - .19	1	2	3	7	
.19 - .20	0	1	5	9	
.20 - .21	0	0	4	2	
.21 - .22	0	0	0	2	
.22 - .23	0	0	0	11	
.23 - .24	0	1	0	4	Grand Total
Total samples	31	72	46	65	214.
Average acidity	.133%	.138	.158	.178	.153
Over 0.18 per cent	3.2%	5.5	26.1	53.8	
Under 0.18 per cent	96.8 %	94.5	73.9	46.2	
Composite sample:					
Acidity	.13%	.12	.14	.15	
Fat	4.00	3.37	5.63	5.21	
Solids-not-fat	8.70	8.51	9.56	9.50	
Total solids	12.70	11.88	15.19	14.71	

These results agree very closely with those obtained by Rice (11), Sommer and Hart (14), Brew (5), and McInerney (9). The average percentage of apparent acidity of the various breeds are comparable to McInerney's, except in the case of the Guernsey, where 0.178 per cent was the average found, while McInerney found 0.139 per cent, with none containing over 0.17 per cent acid.

The apparent acidity results do not agree with those of Fleming and Nair (7), who found no apparent acidity over 0.17 per cent.

It was found that 3.2 per cent of the Ayrshires, and 5.5 per cent of the Holsteins yielded milk higher than 0.18 per cent apparent acidity; while 26.1 per cent of the Jerseys, and 53.8 per cent of the Guernsey samples contained over 0.18 per cent apparent acidity. This would tend to show that some fresh milk titrating over 0.18 per cent might be received at the factory.

The composite sample of all the milk from the Jerseys and Guernseys (taken in February) tested only 0.14 per cent and 0.15 per cent apparent acidity, respectively. The cows giving milk of the higher apparent acidities were yielding only small quantities of milk.

Experiment 2.

APPARENT ACIDITY AND HEAT COAGULATION IN EVAPORATED MILK

Milk secured directly from representative cows of the four principal breeds was evaporated according to the standard procedure outlined on page 20. Two samples from cows in the experimental grade herd are also shown.

In this experiment the milk samples were analyzed for percentage of butterfat, solids-not-fat, total solids, and acidity. When it was necessary to modify the original milk in order to have the butterfat and solids-not-fat in the proper proportion before condensing, cream or skimmilk from that individual sample was used.

The initial acidity, final acidity, source of sample, analyses, and final curd, if any, are shown in Table 2. The samples are arranged according to increasing initial acidity.

Table 2.

APPARENT ACIDITY AND HEAT COAGULATION IN EVAPORATED MILK

NUMBER	INITIAL ACIDITY	FINAL ACIDITY	SOURCE OF SAMPLE	INITIAL TEST			FINAL CURD
				BUTTERFAT	SOLIDS-NOT-FAT	TOTAL SOLIDS	
1	.115%	.29%	H.263	3.28%	8.26%	11.54%	No curd
2	.12	.28	H.263	3.32	8.24	11.56	No curd
3.	.12	.185	Ex.707	6.74	12.06	18.80	Curdy
4	.12	.27	H.comp.	3.37	8.51	11.88	No curd
5	.125	.31	H.263	3.30	8.26	11.56	No curd
6	.13	.245	A.comp.	4.00	8.70	12.70	No curd
7	.14	.345	H.263	2.41	7.52	9.93	Curdy
8	.14	.325	H.251	3.05	8.70	11.75	No curd
9	.14	.30	G.244	3.85	8.75	12.60	No curd
10	.14	.265	G.282	4.78	9.24	14.02	Curdy
11	.14	.27	J.comp.	5.63	9.56	15.19	No curd
12	.15	.29	G.287	6.03	9.28	15.31	No curd
13	.15	.305	G.comp.	5.21	9.50	14.71	No curd
14	.16	.345	G.278	5.35	8.56	13.91	No curd
15	.16	.27	G.282	4.61	9.19	13.80	No curd
16	.17	.345	H.288	2.90	8.97	11.87	No curd
17	.17	.29	Ex.654	6.50	10.50	17.00	Curdy
18	.19	.315	G.271	6.74	9.56	16.30	No curd
19	.215	.42	G.271	6.87	9.62	16.41	Curdy

Summary of Table 2: Average butterfat - 4.63 per cent.
Average solids-not-fat - 9.15 per cent.
Average total solids - 13.78 per cent.
Average initial acidity - .147 per cent.
Average final acidity - .298 per cent.
Percentage coagulating - 26.3 per cent.
Percentage not coagulating-73.7 per cent.

It is interesting to note in this connection that the acidity is concentrated in approximately the same proportion as the milk solids. For this reason, a high apparent acidity need not necessarily result in a high final acidity, as it usually accompanies a high testing milk, and the concentrating ratio is much lower.

The distillate from the milk contained no appreciable acidity, since in three liters one drop of tenth normal alkali rendered it pink to phenolphthalein. This is in accordance with the theory that lactic acid in milk is non-volatile.

Samples number three and seventeen are both exceptionally high in percentage of solids-not-fat and total solids, with normal apparent acidity.

These cows were in an advanced stage of lactation, and were "dried off" shortly after securing these samples.

From these results it is seen that there is no definite relation between the apparent acidity and heat coagulation in sterilizing the evaporated milk.

If these results are examined in relation to the increasing final acidity, no definite point could be established above which the evaporated milk would coagulate.

Experiment 3.

TRUE ACIDITY AND HEAT COAGULATION IN EVAPORATED MILK

In this experiment samples were secured at the receiving platform of the University creamery. The milk represented mixed herd milk from individual patrons, or a composite sample taken from the vat. The acidity in all these samples were characteristic of those received at any condensery, and contained more or less true acidity, depending upon the care the milk had received at the farm or in transit.

The Jersey and Guernsey composite samples were the same ones used in Experiment 2, which had been incubated at room temperature to the acidity noted.

The results are tabulated in Table 3, arranged according to initial acidity, from lowest to highest. The final acidity, source of sample, and condition of the finished evaporated milk are also shown.

Table 3.

TRUE ACIDITY AND HEAT COAGULATION IN EVAPORATED MILK

NUMBER	INITIAL ACIDITY	FINAL ACIDITY	SOURCE	CURD
1	.12%	.25%	Vat	No curd
2	.145	.27	P.23	No curd
3	.15	.31	P.24	Curdy
4	.15	.32	P.24	No curd
5	.15	.27	Vat	No curd
6	.15	.31	P.30	Curdy
7	.155	.295	P.16	No curd
8	.16	.27	P.35	No curd
9	.17	.38	Vat	Curdy
10	.175	.35	Vat	Curdy
11	.175	.32	P.24	Curdy
12	.18	.38	P.30	Curdy
13	.18	.34	Vat	No curd
14	.18	.35	J.comp.	No curd
15	.195	.34	G.comp.	No curd
16	.21	.34	P.30	No curd
Average	.165%	.318%		

Coagulating - 37.5 per cent.
Not coagulating - 62.5 per cent.

No definite relation could be ascertained from this experiment between the initial acidity, which in this case consisted of apparent acidity supplemented by the development of true acidity, and the heat coagulation of the evaporated milk. Milk containing as high as 0.18, 0.195, and even 0.21 per cent acidity made very satisfactory evaporated milk.

From a study of Table 2 and Table 3 it appears that true acidity has a similar effect to apparent acidity upon heat coagulation of evaporated milk. This would be true, of course, only up to the point of initial acidity at which the milk would coagulate on boiling.

Experiment 4.

TITRATABLE ACIDITY AND HEAT COAGULATION IN EVAPORATED MILK

The initial titratable acidity was determined in this experiment without regard as to whether the acidity was apparent or true acidity. The samples were evaporated in the regular manner, and sterilized. The samples

are arranged both as to initial acidity and final acidity, from the lowest to the highest, in each case.

Table 4.

TITRATABLE ACIDITY AND HEAT COAGULATION IN EVAPORATED MILK

Increasing Initial Acidity			Increasing Final Acidity		
INITIAL ACIDITY	FINAL ACIDITY	CURD	FINAL ACIDITY	INITIAL ACIDITY	CURD
.115%	.29%	No curd	.185%	.12%	Curdy
.12	.25	No curd	.245	.13	No curd
.12	.28	No curd	.25	.12	No curd
.12	.185	Curdy	.265	.14	Curdy
.12	.27	No curd	.27	.145	No curd
.125	.31	No curd	.27	.16	No curd
.13	.245	No curd	.27	.15	No curd
.14	.345	Curdy	.27	.12	No curd
.14	.325	No curd	.27	.14	No curd
.14	.30	No curd	.28	.12	No curd
.14	.265	Curdy	.29	.15	No curd
.14	.27	No curd	.29	.115	No curd
.145	.27	No curd	.29	.17	Curdy
.15	.29	No curd	.295	.155	No curd
.15	.31	Curdy	.30	.14	No curd
.15	.32	No curd	.30	.16	No curd
.15	.27	No curd	.305	.15	No curd
.15	.305	No curd	.31	.125	No curd
.15	.31	Curdy	.31	.15	Curdy
.155	.295	No curd	.31	.15	Curdy
.16	.345	No curd	.315	.19	No curd
.16	.30	No curd	.32	.175	Curdy
.16	.27	No curd	.32	.15	No curd
.17	.38	Curdy	.325	.14	No curd
.17	.345	No curd	.34	.21	No curd
.17	.29	Curdy	.34	.195	No curd
.175	.35	Curdy	.34	.18	No curd
.175	.32	Curdy	.345	.16	No curd
.18	.38	No curd	.345	.14	Curdy
.18	.31	Curdy	.345	.17	No curd
.18	.34	No curd	.35	.18	No curd
.19	.315	No curd	.35	.175	Curdy
.195	.34	No curd	.38	.17	Curdy
.21	.34	No curd	.38	.18	Curdy
.215	.42	Curdy	.42	.215	Curdy

Average .155 per cent .307 per cent.

Coagulating - 31.6 per cent.
Not coagulating - 68.4 per cent.

It is evident from an inspection of these data, that while no definite relation exists between the titratable acidity and the heat coagulation in the evaporated milk, the majority of the samples that did coagulate are grouped in the higher acidities, both initial and final.

The average acidity is concentrated approximately two to one. This conforms with the statement previously made that the acidity is concentrated in the same ratio as the milk solids, because the average milk of 3.9 per cent butterfat, 8.55 per cent solids-not-fat, and 12.75 per cent total solids, concentrated two to one yields evaporated milk of the United States Government standard of 7.80 per cent butterfat, 17.70 per cent solids-not-fat, and 25.50 per cent total solids, providing there is no serious loss in condensing.

It appears, therefore, that the condensery would not be justified in rejecting the milk for evaporated milk purposes on the basis of the titratable acidity alone. However, more difficulty is likely to be encountered in the milk of higher titratable acidity, as shown by these data.

Experiment 5.

MIXTURE OF NORMAL AND SOUR MILK, AND INCUBATING NORMAL MILK

Since milk of high titratable acidity is often mixed with milk of lower acidity at the receiving platform, an experiment was undertaken to find out the effect of such mixing on the heat coagulation of the evaporated milk.

Two portions of milk samples of low acidity were incubated at room temperature to varying degrees of acidity. They were then mixed with the original sample (held at 32° to 35° Fahrenheit) in varying proportions to produce different degrees of acidity. These samples were then evaporated in the regular manner, and the effect on the finished product noted.

Similar samples were incubated to a high acidity, but not to the point of souring, and evaporated in the regular manner.

The results are shown in Table 5.

Table 5.

MIXTURE OF NORMAL AND SOUR MILK, AND INCUBATING NORMAL MILK

NUMBER	INITIAL ACIDITY OF THE MILK	ACIDITY AFTER INCUBATION	SOUR MILK ADDED	ACIDITY BEFORE CONDENSING	ACIDITY AFTER CONDENSING	CURD
1	.13%	.47%	5%	.15%	.31%	Curdy
2	.13	.28	33	.18	.38	Curdy
3	.14	.18	none	.18	.35	No curd
4	.15	.195	none	.195	.34	No curd
5	.14	.21	none	.21	.34	No curd
6	.14	.23	(coagulated on boiling)			

The addition of only five per cent of very sour curdled milk caused the entire batch to coagulate in sterilizing. A similar result was secured by adding a larger quantity, thirty three per cent, of a slightly sour milk.

When the original sample was condensed before it had produced enough acidity to coagulate on boiling, the finished product showed no curdiness.

This would tend to show that milk of an acidity insufficient to cause coagulation on boiling might be mixed with normal milk without increasing appreciably the tendency to coagulate in the sterilizer. However, it would act as a "starter" for the development of acidity, and would be very objectionable from that viewpoint. The addition of a little curdled milk tends to cause coagulation in the sterilizer.

Experiment 6.

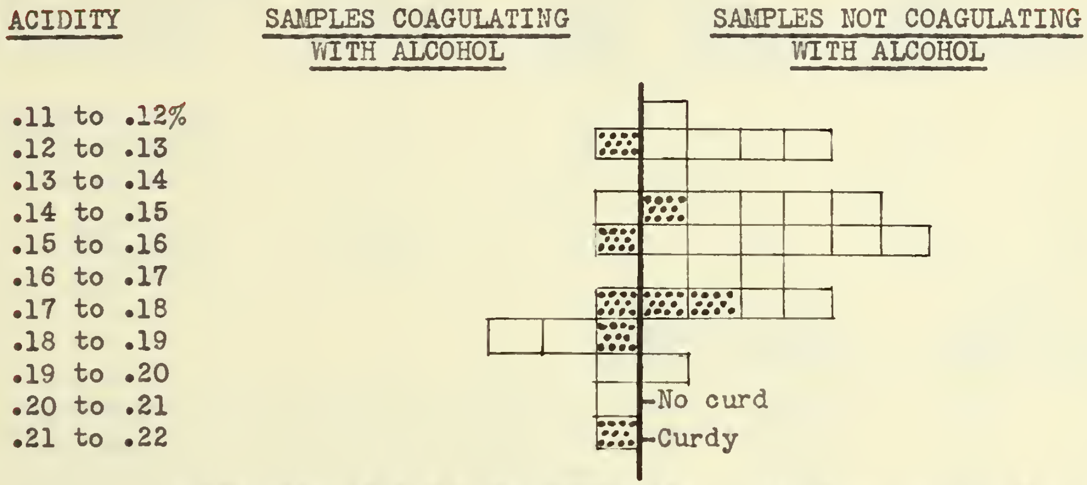
COMPARISON OF TITRATABLE ACIDITY AND THE ALCOHOL TEST

In connection with titratable acidity, alcohol tests were made on the milk, as outlined under "procedure" on page 19.

Figure 5 shows the relation of titratable acidity and the alcohol test to heat coagulation in evaporated milk. Samples to the left of the heavy vertical center line coagulated with seventy per cent alcohol; while those to the right did not coagulate with the alcohol. The dotted units indicate that the evaporated milk coagulated on heating in the sterilizer, while the clear units showed no curd formation.

Figure 5

THE RELATION OF TITRATABLE ACIDITY AND THE ALCOHOL TEST TO HEAT COAGULATION IN THE EVAPORATED MILK



These results are also summarized in Table 6, Comparison of acidity and alcohol tests, and Table 7, Relative value of acidity and alcohol tests.

Table 6.

COMPARISON OF ACIDITY AND ALCOHOL TESTS

ACIDITY	COAGULATES WITH 75% ALCOHOL			NO COAGULATION WITH 75% ALCOHOL		
	TOTAL SAMPLES	EFFECT OF STERILIZATION		TOTAL SAMPLES	EFFECT OF STERILIZATION	
		CURDY	NOT CURDY		CURDY	NOT CURDY
.11 to .12%	0	0	0	1	0	1
.12 to .13	1	1	0	4	0	4
.13 to .14	0	0	0	1	0	1
.14 to .15	1	0	1	5	1	4
.15 to .16	1	1	0	6	0	6
.16 to .17	0	0	0	3	0	3
.17 to .18	1	1	0	4	2	2
.18 to .19	3	1	2	0	0	0
.19 to .20	1	0	1	1	0	1
.20 to .21	1	0	1	0	0	0
.21 to .22	1	1	0	0	0	0
Totals	10	5	5	25	3	22

Table 7.

RELATIVE VALUE OF ACIDITY AND ALCOHOL TESTS

KIND OF EVAPORATED MILK SAMPLES	NUMBER OF SAMPLES	ALCOHOL TEST TIMES CORRECT	ALCOHOL TEST TIMES INCORRECT	ACIDITY TEST TIMES CORRECT	ACIDITY TEST TIMES INCORRECT
Satisfactory	27	22	5	22	5
Unsatisfactory	8	5	3	2	6
Total	35	27	8	24	11
Per cent	100%	77.2%	22.8%	68.4%	31.6%

	ALCOHOL TEST NUMBER	ALCOHOL TEST PER CENT	ACIDITY TEST NUMBER	ACIDITY TEST PER CENT
Satisfactory milk samples retained -	22	81.5%	22	81.5%
Satisfactory milk samples rejected -	5	18.5	5	18.5
Unsatisfactory milk samples retained-	3	37.5	6	75.0
Unsatisfactory milk samples rejected-	5	62.5	2	25.0

This comparison between the titratable acidity and the alcohol test tends to show that the titratable acidity would have rejected as many of the satisfactory samples as did the alcohol tests; but that more of the unsatisfactory samples would have been rejected by the alcohol tests as compared to the acidity tests.

As an index of the coagulability, it is apparent that neither the titratable acidity nor the alcohol test are reliable, and that some other means should be evolved for determining the quality of milk intended for evaporated milk.

V. SUMMARY AND CONCLUSIONS

SUMMARY

In the manufacture of evaporated milk difficulty is often encountered in that the milk coagulated on heating in the sterilizer. This has been found to be due to a number of factors, some of which are under the control of the manufacturer, while others are at present impossible of control by the manufacturer of evaporated milk.

The factors under the control of the manufacturer are: the

temperature in the forewarmer; concentration; pressure in the homogenizer; addition of sodium bicarbonate; temperature and time in sterilizing; shaking; and titratable acidity.

The titratable acidity is usually controlled by rejecting milk delivered to the factory above a certain specified standard, as 0.18 per cent. Milk has been found to contain apparent acidity, varying from 0.10 to 0.25 per cent, according to breeds and individual cows. No definite relation has been established between the apparent acidity and heat coagulation in evaporated milk.

The composition and balance of the milk salts and the hydrogen-ion concentration are not under the control of the manufacturer of evaporated milk. Rogers, Deysher and Evans (13) showed that the composition of the salts of milk and the hydrogen-ion concentration have no direct influence on the temperature at which the evaporated milk curdles, but that any possible importance may be obscured by other factors.

Determination of apparent acidity showed that variation exists between individual cows and different breeds. The higher apparent acidities are usually associated with the higher testing milk. Twenty six per cent of the Jersey and fifty three per cent of the Guernsey samples showed 0.18 per cent apparent acidity, or higher; as compared to three and six per cent, respectively, of the Ayrshire and Holstein samples.

There appears to be little differentiation between titratable, apparent, and real acidity in their lack of direct correlation to the heat coagulation of evaporated milk.

The alcohol test and the titratable acidity test rejected the same proportion of samples that turned out to be satisfactory, but the alcohol test rejected a larger percentage of the unsatisfactory milk samples.

CONCLUSIONS

1. Apparent acidity in milk varies from about 0.10 to 0.25 per cent, according to individual and breeds. The higher apparent acidity is usually found in the higher testing milk, as typified by the Jerseys and the Guernseys.

2. No direct relation exists between apparent acidity and heat coagulation in evaporated milk.

3. There is no definite effect of titratable acidity upon heat coagulation in evaporated milk, and condenseries are not justified in rejecting milk on the basis of the titratable acidity alone.

4. The alcohol test is not a reliable index of the coagulability of the milk in the manufacture of evaporated milk, but rejects a larger percentage of unsatisfactory samples than the titratable acidity test.

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